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(12) DESCRIPTION OF AN INVENTION

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(54) METHOD OF CONTINUOUS FIBER MAKING FROM THE MELT OF BASALT ROCKS

Application: for production of chemically stable/resistant, high-temperature filters, fabrics, construction goods. Essence of the invention: basalt rocks in fraction of 5-15mm. and acidity module 4.7-6.5 are melted without corrective additive with the temperature in the furnace 1500-1600°C in oxidizing medium. With the melt temperature rise up to 1600°C its amorphism degree increases. $Fe_2O_3 : FeO$ ratio is not less than 1.2 and total content of ferric oxides – 8-12%. Fibers production is conducted at temperature 1310-1370°C and viscosity 12-85 Pa·sec in the ratio of $5 < \frac{\eta}{d_\Phi^2} < 25$. Limit temperature interval of the fiber making – not less than 70°C, temperature of the upper limit of crystallization – not more than 1250°C [table 3].

DESCRIPTION OF AN INVENTION.

Invention is related to the continuous fibers production from the basalt rocks melts, which can be used for making chemically resistant, high-temperature filters, fabrics as facing materials in production of stitched heat-insulating sound absorbing wares, for making various composites for construction application, reinforcing of polymer bindings and cement-plaster bindings, and being a substitute for asbestos, e.g. in braking blocks.

The known method of making fiber from rocks includes breaking up, melting of the rock and making fiber [1].

Disadvantage of this method is using of only one type of raw material – pyroxene porphyrite from the Khavchozersk deposit in Karelia, and relatively low strength of the fiber.

Most similar is the method of making fibers from rock melts, using basalt rocks [2]. In accordance with this method a finely grounded basalt is used and fiber making is conducted at the temperature of the melt 1200-1300°C and 10 Pa·sec.

Disadvantage of this method is instability of the process and a narrow interval for continuous fibers making, because in the mentioned temperature range an initial crystallization of the melt occurs, which

cause breakage of threads. Practice reveals that temperature of making should be for not less than 80°C higher than the temperature of the upper crystallization limit.

Moreover, at the viscosity of 10 Pa·sec and fixed content of ferric oxides (15-20) a good wetting ability of the melt towards platinum-rhodium alloy is registered and a consequent flowing of the draw plate area. It is necessary to mention that given melting temperatures (1200-1350°C) are low, because such a melt contains unfused particles.

Furthermore, in this method only basalt is used, though class of basalt rocks includes a quantity of rocks, characterized by the different texture-structure peculiarities and variety of the chemical composition.

This invention purpose is to extend the temperature range of the fiber making, improve strength and temperature resistance of the fiber; and expand the class of rocks used – from basic to medium composition.

A fixed purpose of the supposed invention is reached by alteration of the processing parameters. Rocks in fraction of 5-15mm. are melted without corrective additive with the temperature in the furnace 1500-1600°C in oxidizing medium. With the melt temperature rise up to 1600°C its amorphism degree increases, i.e. a full change from the crystalline state into the vitreous state. This cause rise of the breaking point at tension [table 1]. Amorphism degree of the fiber was measured by two methods: IR spectrography in range 400-4000cm^{-1} by absorption bands and micro-photography method. A close fit of results acquired by these methods was noticed. Amorphism degree and strength of continuous fibers, made from basalt rock melts, at different temperatures in the furnace are given in the table 1.

From the table 1 it is obvious that considerable rise of the amorphism degree and strength parameter occurs after the melt stay in the temperature range 1500-1600°C; further temperature rise is inexpedient both economically and due to the melt destruction (destruction of individual components).

Basalt melting is conducted in the oxidizing medium in gas-heated furnace. Air excess coefficient while supplied for combustion should be in the range 1.15-1.25. This is required because of the following.

Basalt rocks contain iron in two forms: Fe^{2+}; Fe^{3+}. Usually ferrous iron prevails in the rock. When rock is melted in the oxidizing medium, an oxidation reaction occurs as follows:

$$4FeO + O_2 \rightarrow 2Fe_2O_3$$

Content of Fe_2O_3 in the fiber increases and ratio $Fe_2O_3 : FeO$ of not less than 1.2 is reached. Such a ratio of iron oxides secures a smaller loss of fibers strength under high temperature operation, when fibers crystallization occurs. It results from the formation of the fine-grained haematite, around which pyroxenes are crystallized. In case of $Fe_2O_3 : FeO < 1.2$ a macrocrystalline magnetite is formed in the first phase (at lower temperatures than haematite) and then pyroxenes. A retained strength of fibers, when haematite is crystallized, is higher, especially at high temperatures [table 2].

Note: initial strength of fibers with different $Fe_2O_3 : FeO$ ratio is equal, MPa: 2020, 2140, 2300, 2500 accordingly.

Analysis of the table data reveals an obvious effect of smaller strength loss of fibers with $Fe_2O_3 : FeO$ ratio, starting from 500°C and higher. Thus, a retained strength of fibers at the temperature 600°C with $Fe_2O_3 : FeO$ – 1 makes 18.3%, whereas with $Fe_2O_3 : FeO$ – 1.2 and temperature 700°C the retained strength is in the range of 20.1-28.6%. For fibers with $Fe_2O_3 : FeO$ – 1.2 at the temperature 750°C a retained strength is 9.5-12.2% while fibers with $Fe_2O_3 : FeO$ – 1 are fully destructed.

Thus, basalt rocks' melting under the proposed conditions (temperature in the furnace 1500-1600 ° C and oxidizing medium) makes it possible to increase an initial strength of fibers, comparing with the prototype (melting temperature 1350°C), in 1.4-1.6 times. Securing an optimal $Fe_2O_3 : FeO$ ratio (not less than 1.2) makes it possible to increase the fibers application temperature for more than 100°C (up to 800°C).

To produce a continuous fiber basalt rocks of the following chemical compound are suitable, mass: SiO_2–47-57; Al_2O_3–13-18; TiO_2–1.2-2.0; $Fe_2O_3 + FeO$–8-12; CaO–6-12; MgO–3-8; $K_2O + Na_2O$–3-6.

Acidity module (M_K) is calculated by the formula $M_K = \frac{SiO_2 + Al_2O_3}{CaO + MgO}$ and has to be in limits of 4.7-6.5 and $Fe_2O_3 + FeO$–8-12%.

To extend a temperature interval of fiber making ($TI_{B.B.}$) important factors are viscosity (η) and temperature of the upper limit of the melt crystallization ($T_{B.П.K.}$). It is also required to consider a correlation between viscosity and square diameter of the draw plate ($d_Ф$): $\frac{\eta}{d_Ф^2}$ that determines stability and continuity of the fiber forming process.

Table 3 indicates the dependence of the working interval.

Diameter of the draw plate was altered from 1.6 to 2.2mm. depending on the viscosity of the melt and the rock type. In case of $\frac{\eta}{d_Ф^2}$ more than 5, but less than 25 in the working temperature interval of 1310-1370°C (temperature on the draw plate) a limit temperature interval of fiber making (difference between the upper and lower limit temperatures of the fiber forming process) extends and makes 70-110°C. This parameter for the prototype is 40°C. Temperature of the upper crystallization limit ($T_{B.П.K.}$) is not more than 1250°C (prototype – 1275°C). Viscosity at the working temperature interval is in the range of 12-85 Pa·sec.

Mentioned above parameters of the continuous fibers production process were ascertained for the wide range of basalt rocks from basic to medium composition (basalts, basanites, gabbro, basalt, diabase, andesite porphyrites, andesite-basalts etc.) which are characterized by the different texture-structure peculiarities and variety of the chemical composition.

The proposed method is planned to be applied at the basalt fiber production lines under construction.

SUBJECT OF AN INVENTION

Method of continuous fiber production from the basalt rocks melt, including rocks shattering, melting and making fiber through the draw plates, **which differs** in the supplied basalt rock with the acidity module 4.7-6.5, melting is conducted in the oxidizing medium at the temperature in the furnace 1500-1600°C and fiber making interval is 1310-1370°C with the correlation between viscosity of the melt and square diameter of the draw plate equal to $5 < \frac{\eta}{d_Ф^2} < 25$.

Table 1

Temperature of the melt	Diameter of the fiber	Amorphism degree, %	Tensile strength limit, MPa
1300	8.3	25.3	1600
1350	8.4	26.5	1620
1400	8.6	35.0	1870
1450	8.5	50.0	2010
1500	8.7	80.0	2300
1550	8.6	95.0	2500
1600	8.7	96.0	2550

Table 2

Temperature of the fibers heat treatment	Retained strength of fibers, % with following $Fe_2O_3 : FeO$ ratio			
	1	1.2	1.5	1.7
20	100	100	100	100
100	100	100	100	100
200	100	100	100	100
300	98.0	98.2	98.7	100
400	75.2	85.3	88.7	90.0
500	39.0	56.2	58.9	65.0
600	18.3	30.0	32.4	38.8
700	0	20.1	25.1	28.6
750	0	9.5	10.2	12.2
800	0	0	0	0

Table 3

Temperature, °C				$T_{B.П.К.}$, °C	$TI_{B.B.}$, °C
1310		1370			
η, $Pa \cdot sec$	$\frac{\eta}{d_\Phi^2}$, $\frac{Pa \cdot sec}{mm^2}$	η, $Pa \cdot sec$	$\frac{\eta}{d_\Phi^2}$, $\frac{Pa \cdot sec}{mm^2}$		
32.2	9.9	17.8	5.5	1240	70
38.3	11.8	19.0	5.9	1230	100
41.6	12.8	23.5	7.2	1240	80
52.0	16.0	26.8	8.3	1200	110
65.6	16.4	36.0	9.0	1250	70
74.2	18.5	36.4	9.1	1250	70
81.0	20.2	44.6	11.1	1241	70
Prototype					
19.8	6.1	11.2	3.5	1275	40
Impossible to measure viscosity		275	56.8	1250	20